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<b>14. ABSTRACT</b>  This report results from a contract tasking Kharkov Institute of Physics and Technology as follows: The current project is directed toward developing the curvalence arc plasma source of carbon and metal plasma for producing tribological films. This includes plasma source design, study, and optimization to operate with graphite, aluminum, and zirconium targets, and to produce low friction and hard diamond-like carbon (DLC), alumina, and zirconia films. A study of plasma properties, including ionization state, density, and energy distributions will be performed. The results will be used to manufacture a demonstration system with an optimized plasma source by the KIPT. This system will be used by the KIPT to study plasma characteristics in tribological material synthesis and prepare samples of tribological films for evaluation at the Air Force Research Laboratory, Wright-Patterson Air Force Base (AFRL, WPAFB), Ohio. The system will be then transferred to AFRL, WPAFB together with required power supplies, control modules, and design documentation for installation, testing, and on-site technology demonstration. Specifically, the use of the technology in production of DLC films with a high percentage of sp3 electronic hybridization, synthesis of cubic BN, deposition of crystalline metals, oxides, carbides, and nitrides at low substrate temperatures, and production of nanocomposite materials made of nanocrystalline and amorphous phases will be the focus of the seminars.					
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Final Report

Project P-069

**Magneto-Optically Guided and Fully Ionized Metal / Carbon Arc Plasma  
Source for Thin Film Coatings to Control, Wear, and Fretting**

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NSC KIPT

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## INTRODUCTION

Ion beam plasma sources are currently widely used in deposition of thin films for various applications: electronics, optics, magneto-storage, tribology, *etc.* The first ion-beam plasma sources were designed for space ion-thruster technology, *e.g.* correction engines for satellites. It was then recognized that they could be beneficially used for surface cleaning, reconstruction, and structure control in thin-film synthesis by delivering high-energy ions to the condensation surface [1]. The benefit of these sources for growing protective films to counteract friction, wear, fretting, improve adhesion, prevent fatigue cracks, and corrosion damage was highlighted in Ref. [1].

Ion-beam sources operated on gas precursors do not typically provide direct film growth. When they are used with inert gases, only surface cleaning and structural reconstruction can be performed. When they are used with nitrogen, oxygen, and hydrogen, mainly film chemical doping is performed due to a low density of atoms in the generated plasma. One exception is the sources operated on hydrocarbon gases, which are used to grow hydrogenated diamond-like carbon (H:DLC). This was first demonstrated by Aisenberg and Chabot, using a source developed using ion-thruster technology under a contract with NASA [2].

Ion-beam technologies with plasma generation from gases have a low flux density and are not suitable for the production of pure metallic ion beams. It was also recognized, that the presence of hydrogen leads to deterioration of film mechanical properties, such as hardness and wear resistance, due to the hydrogen termination of diamond-like  $sp^3$  hybridization links within amorphous networks. Therefore, there was a need for a hydrogen-free DLC ( $\leq 1$  atomic % of hydrogen). Currently, there are two technologies, which can provide such films. One is carbon plasma generation and film growth using laser ablation of graphite, where most recent advances for tribological applications were reported by scientists of AFRL, WPAFB [3]. Another is generation of carbon plasma using vacuum arc technology invented in the KIPT (see US patents on this technology #3,793, 179 and #3,783,231).

The vacuum-arc discharge is characterized by high power and efficient generation of a highly ionised plasma with energy about some tens eV. The fraction of ionic current extracted from the plasma is about 0.1 of the arc current (for example, at a current 100 A the ion current is 10 A) and the coating deposition rate can approach 40  $\mu\text{m/h}$ . However, a vacuum arc plasma generates a large amount of droplets, which deteriorate the properties of growing films. In order to overcome this problem, a magnetic filter for extraction of metal ions from vacuum arc plasmas and guiding them toward the substrate was invented in the KIPT by Aksenov and Belous and continued to be developed by Strel'nitskij, Ukraine [4-18].

The KIPT filter design is currently recognized to be the most successful among other designs discussed within international scientific community [19]. It separates heavy macroscopic particles and ions passing along curvilinear plasmaguiding channel with crossed electrical and magnetic fields. Unlike other methods, this provides more complete filtering of plasma from droplets (or hard fragment) of cathode material. However, it may lead to a large loss of the plasma flow on the walls of an extended (above 1 m) and narrow plasma guide with a 90° bend. Besides, the output of a filtered plasma flow has a small cross-section and a large ion density gradient.

In the recent literature, there are many publications on various reproductions and studies of curvilinear filters, designed after the work in the KIPT [20, 21]. However, the output parameters (current density, flux uniformity) of the investigated systems are similar to that obtained by Aksenov and Strel'nitskij earlier.

At the same time, our experience indicates that the output parameters of curvilinear filters are far from their theoretical maximum. Some preliminary data indicate the possibility to increase ion transport efficiency by several times. This will ensure the increased deposition rate, an improved coating uniformity, and a better coating microstructure control, required for advanced tribological film growth. Dense, uniform, and energy controlled beam of metal ions will open new possibilities DLC films with high  $sp^3$  interatomic bonding content, deposition of nanocrystalline oxides, nitrides, and carbides at low substrate temperatures, production of nanocomposite coatings. This may also open path to a low temperature synthesis of such technologically important materials as cubic BN or  $\beta$ -C<sub>3</sub>N<sub>4</sub>.

The main objectives of this project were: design of an improved metal/carbon ion-beam source; basic study of vacuum arc plasma transport in a crossed electric and magnetic fields; plasma filter optimisation; assembly of a demonstration system; preparation and characterisation of metal plasma to be used for the growth of transition metal oxides, nitrides, or carbides in wear protective coatings.

The main results of experimental researches of the vacuum-arc plasma source with curvilinear electromagnetic system for clearing of the macroparticles from plasma which has been carried out during the first year of this project, may be formulated as follows.

The magnification of linear dimensions of a cross-section of the system under consideration (in explored variant – approximately up to 200... 300 mm) at small aspect relation ( $R/a \approx 1,3$ ), that is close to the minimal possible value ( $R/a = 1$ ), promotes substantial increase of throughput capacity of the system. In rectilinear input (anodic) part of the system, as the previous researches have shown, the plasma losses are negligible. However, it concerns only to plasma of metals which is transported in high vacuum or at presence of gases, at interaction with which

plasma condensing does not form compounds with a high resistance. In other cases (for example, at forming streams of carbon plasma) it is necessary to take special measures to ensure stable arcing. In our case there was an anodic insert. With its aid the problem of burning of an arc was solved. But thus not less than a third of plasma stream was lost at the insert.

The main part of losses pertains to the curvilinear part of the plasma guiding channel. Plasma losses here are stipulated by diffusion of particles across magnetic field toward the walls of the plasma duct. Besides the part of the plasma stream drifts on the walls under reaction of crossed magnetic field and polarization electric fields arisen in plasma because of inhomogeneity and curvature of the magnetic field (so-called gradient and centrifugal drifts).

It was found, that the drift losses may be reduced noticeably by local adjusting of the magnetic field by an angle displacement of the magnetic coil in the curvilinear part of the plasma guiding channel.

It is revealed also, that the considerable decrease of the drift losses may be reached applying the negative potential to a part of the curvilinear duct wall near to its output aperture from the side, counter to  $[R \times H]$  direction or in  $R$  direction at the positive bias potential on the plasma duct as a whole.

The amount of losses, irrespective of their origin, decreases, and the ion current on the exit of the curvilinear part of the plasma guiding channel increases when the positive duct biasing potential rises. In explored variant of the system its maximal throughput capacity was detected at the bias voltage  $+25 \dots +30$  V. In the mode of "partly negative" duct (see the previous paragraph) maximal values of throughput capacity of the system are reached. Thus, as against traditional condition, the output ion current increases, not peaking, with rise of the positive bias up to 50 V.

The shift of a main body of the stream (its "core") in the plane of symmetry of the duct happens in a streamline of guiding magnetic field lines which originate at the active surface of the cathode. In explored system these lines and, hence, the guided plasma stream shifted toward the center of curvature of the field while the usual concept about a motion of plasma along a magnetic field at  $\text{grad } H \neq 0$  orders the backward direction of the shift.

The obtained results were used when developing the concept of the new vacuum-arc system for shaping of streams of plasmas free from macroparticles. The designed system contains the vacuum-arc plasma source with magnetic stabilization of a cathode spot drift zone and with a magnetic focusing of the plasma stream generated by this spot. The cylindrical anode with focusing coils fulfills also functions of the input section of the  $\Gamma$ -shaped duct of the new system. Such duct with magnetic coils ensures turn of a plasma stream on  $90^\circ$ . Thus the key opportunity of diminution of the aspect relation  $R/a$  up to extreme small value close to unit is

achieved. The abbreviated drawing of the system is shown in fig. 19 (see Ref. [22]). As against  $\Gamma$ -type variant, here the plasma duct contains also a trap for macroparticles. This trap looks like a cylindrical container. Its side walls are prolongation of the input section of  $\Gamma$ -shaped plasma duct so the construction as a whole gains the T-shaped form. An output section of the plasma duct – its construction and dimensions – are defined by the construction of the vacuum chamber of installation to be used for experimental researches of the new system. The rigid demands to the construction and dimensions of the output section have stipulated the choice of dimensions of the rest of parts of the system.

In the system construction overabundant facilities of forming of magnetic fields of different geometry and intensity are included. It was supposed that the final selection of the magnetic coils system would be realized using results of computation and test of the experimental sample of the plasma filter. Preliminary computer calculations of MPs trajectories and magnetic field topography in plasma guiding channel of the new system have been performed. The rough estimate of a degree of the plasma stream filtering in the system depending on geometry of interior surfaces of the plasma guiding channel have been accomplished with the aid of the data received.

All the above-mentioned results are described in detail in the annual report [22], and the results of further investigations are presented in the present final report. Here we described the design of the vacuum-arc filtered plasma source developed with taking into account the experimental data obtained during the first year of activity under the project. The report includes the results of studies of the influence of some structural anode elements as well as energizing schemes of magnetic coils in the plasma source on the stability of vacuum-arc ignition and burning; the data on plasma losses minimization and increasing the efficiency of plasma transport along the filtering channel; the results of measuring distribution of the density of a filtered plasma stream across its cross-section at the filter exit; the summarized results of comparative investigations and tests of DLC and  $CN_x$  coatings obtained by the vacuum-arc and laser methods in AFRL, WPAFB.

## 2. EXPERIMENT AND MEASUREMENT METHODOLOGY

The schematic drawing of the plasma source being studied is presented in Fig.1. Conceptually this source is analogous to the source with T-shaped filter described earlier in [22]. A vacuum-arc plasma generator of the device under consideration comprises a cathode unit (1) and a water-cooled cylindrical anode (2). The cathode unit is assembled of a dismountable body, a water-cooled cathode in the form of a truncated cone, an igniter and a coil of stabilizing magnetic field  $L_{st}$ . The layout and the operating principle are described in the Ref. [23, 24]. The diameter of the major cathode base is 64 mm, the initial height is 40 mm. The inner anode diameter is 180 mm, the length is 270 mm. We have studied the plasma source having an anode with an insert and without insert. The anode insert is a grid made from parallel tungsten rods. The rod diameter is 4 mm, the gaps between rods are 16 mm. Thus, the grid transparency was 80%. The insert from tungsten rods was used in experiments with a titanium cathode. In the cases when the cathode material was graphite, we used the anode insert in form of a set of plates from special composite carbon material (carbon cloth impregnated with pyrocarbon). The cross-section of plates is  $3 \times 20 \text{ mm}^2$ , the gaps between the plates are 16 mm. The grid transparency is 84%.

The ion current at the anode exit was measured with the help of a flat stainless steel collector - disk. The disk diameter is 167 mm. The collector was placed at a distance of 330 mm from the working edge of the cathode. In the course of measurements the negative (relatively to the grounded anode) potential of 70 V was applied to the collector.

The T-shaped plasma duct comprises an input (4) and an output (5) parts in the form of rectilinear tubes joined at a right angle. The input part of the plasma duct beyond the site of joint with the output part transforms into the trap of macroparticles being a cylindrical container (6) on the bottom of which a set of inclined plates (7) is placed that prevents the ricocheting of macroparticles towards the system exit. For the same purpose, in the output part of the plasma guide there are installed an assembly of plates in the form of half-rings (8) and an assembly of flat rings (9). The transporting magnetic field in the plasma guiding channel of the filter is created by the coils  $L_1 - L_3$  and  $L_7 - L_9$ . The coils  $L_4 - L_6$  are designed for the magnetic field correction in the region of the plasma guiding channel bended at  $90^\circ$ . The variants of correcting coil connection are shown in Fig.2. An additional output section of the plasma duct (10) is used for joining the above-mentioned filter with a process chamber and for transporting the plasma flow to the substrate. To generate transporting and deflecting magnetic fields there are coils  $L_{11}$  and  $L_{12}$  arranged in a casing of the additional output section of the plasma duct. The vacuum valve 11 and the coil  $L_{10}$  shown in the drawing were not available in our experiments.



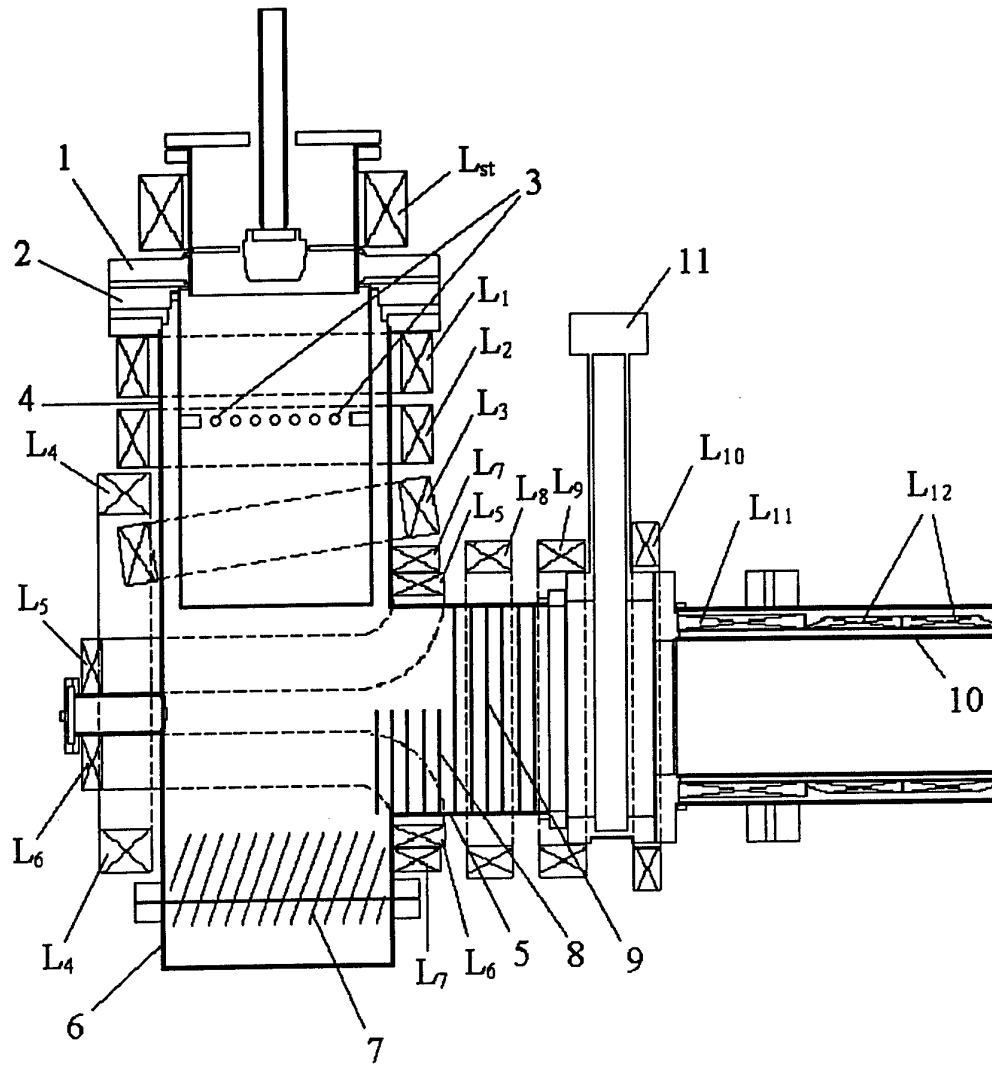


Fig. 1. Filtered vacuum-arc plasma source.

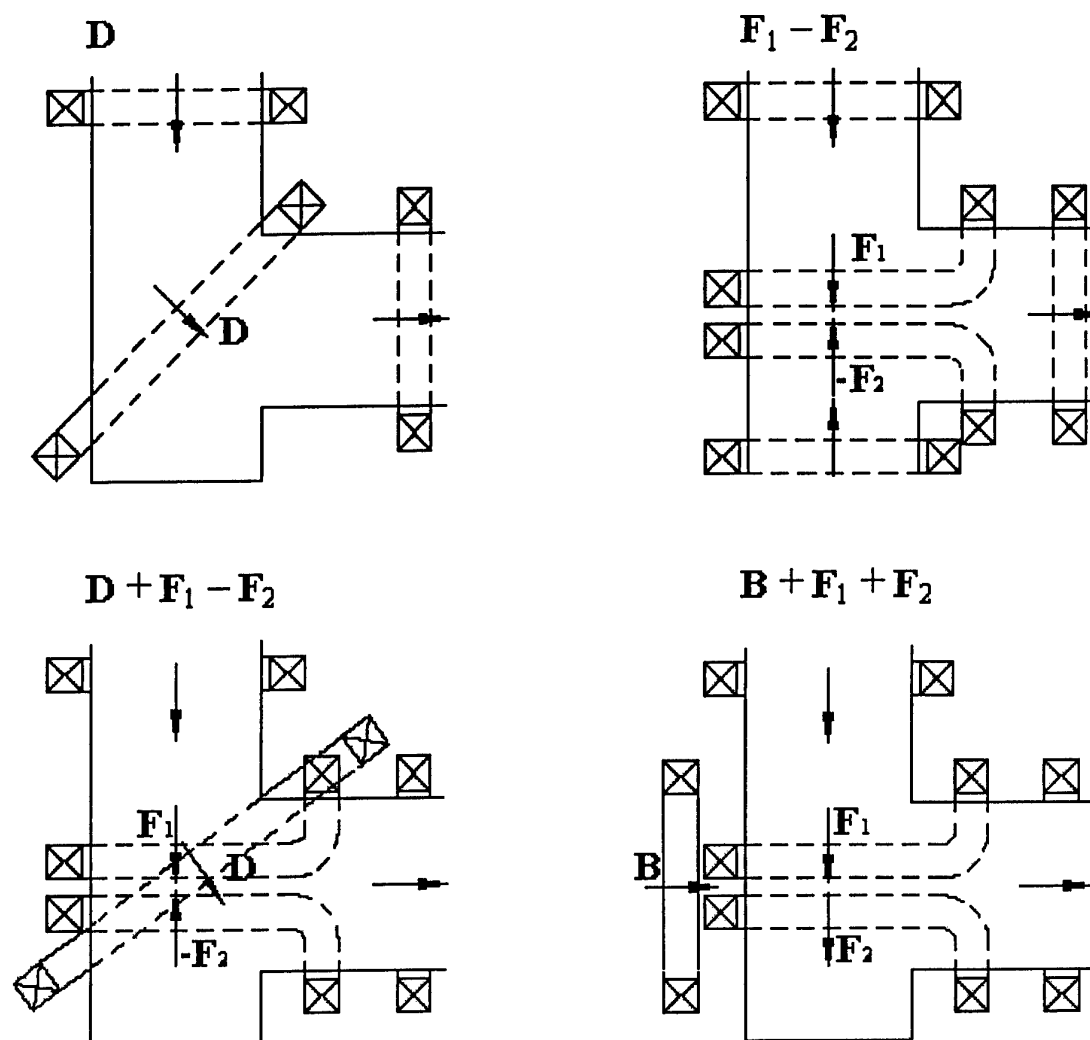


Fig. 2. Schemes of the curvilinear magnetic field correction.

The power for the vacuum arc was supplied from the dc current source via the ballast resistor (0.4 Ohm). The no-load voltage of the source was 120 V. The power supply of magnetic coils was performed from another source with separated exits. Parameters of coils and a power of their current sources provide the creation of magnetic fields on the plasma guide axes up to 150 Oe, during short time - up to 250 Oe.

The parameters of correcting coils are the following:

- Diagonal coil (Fig.2): average diameter  $d = 470$  mm, number of turns  $n = 172$ , (6.5 Oe/A);
- Figured coils ( $L_5, L_6$ ):  $d = 380$  mm,  $n = 100$ , (4.0e/A);
- Edge coil ( $L_4$ ):  $d = 470$  mm,  $n = 100$ , (3.67 Oe/A).

The plasma duct of the filter in the described variant was insulated from the anode and the grounded chamber of the device. During operation of the source the plasma duct was either under the floating potential or under the controlled positive bias voltage applied from the special source.

As it follows from Fig.1, in the plasma source under consideration, unlike the conceptual version of the source described in report [22], the anode is placed inside the input part of the plasma duct and overlaps fully the inner surface of the latter. Thus, the anode 2 plays the role of the input arm of the T-shaped plasma duct.

The ion current at the exit of the curvilinear part of the plasma guiding duct was measured with the help of the flat titanium collector in the form of a disk of 180 mm in diameter (disk-collector). The collector was disposed in the output part of the plasma duct 5 in the region of the coil  $L_9$ . The ion current of the plasma beam at the system exit was measured with the help of the flat collector in the quadrate form (quadrate – collector) (250x250 mm<sup>2</sup>), installed in the chamber at a distance of 110 mm from the exit edge of the additional section of the plasma duct 10.

The character of plasma density distribution in the cross-section of the output flow was determined by the distribution of the condensate (DLC) thickness deposited from the carbon plasma on the flat substrate surface. The technique of measuring the thickness is described in Ref. [22].

The angular distribution of the plasma ion flow in the region of plasma guiding duct bending at 90° (between the input (4) and output (5) plasma duct sections) was measured with the help of the flat probe in the form of a plate of 110 x 220 mm<sup>2</sup>. The probe was mounted in parallel to the walls of the plasma duct input section at a distance of 100 mm from the axis of the latter. Therewith a possibility to rotate the probe around this axis and to fix it in any position specified by the angle  $\alpha$  from 0° to 360° (Fig.3c) was provided. The angle  $\alpha = 180^\circ$  corresponded to the direction from the center toward the exit opening of the filter. The probe can

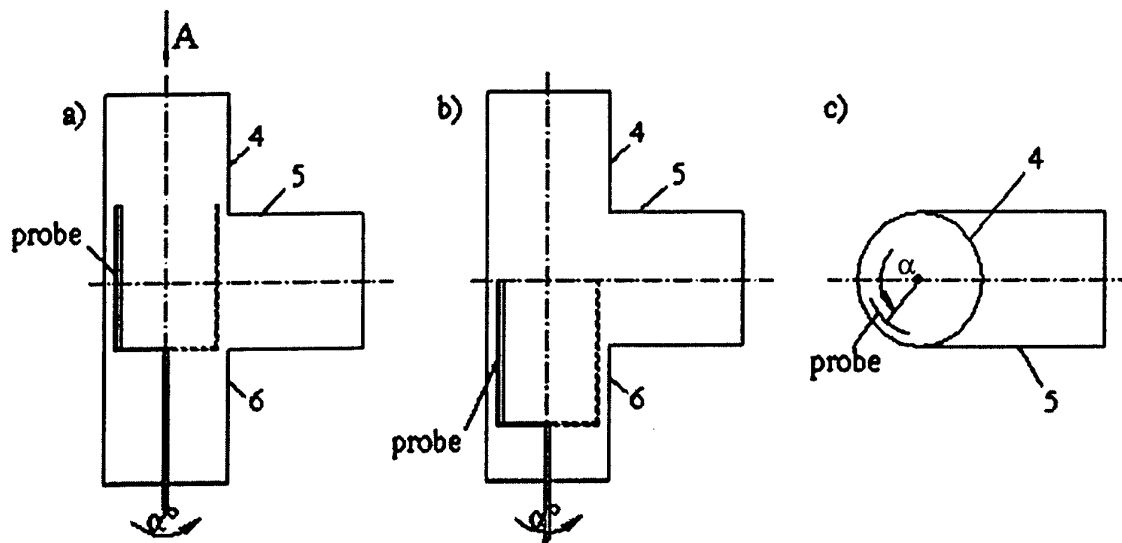


Fig. 3. Schematic of the turning probe position.

- (a) – the probe overlaps the output duct aperture in its height completely;
- (b) – the probe overlaps the half of the duct aperture;
- (c) – top view (along the arrow A).

be displaced in parallel to the axis of the input plasma duct. The measurements were performed with the probe installed so that by the length it overlaps the input aperture of the output section (5) of the plasma duct (Fig.3a) either fully or only its half-part (Fig. 3b) on the the side of the trap 6.

Distribution of the longitudinal component of the magnetic field along the axis of the input part of the plasma duct was measured with the help of the Hall magnetometer.

### 3. RESULTS AND DISCUSSION.

#### 3.1. The ion current at the anode output

The ion current was measured with energized coils creating the transporting magnetic field in the plasma filter. So, the influence of the total filter magnetic field on the vacuum-arc generator operation was taken into account. The cathode material was titanium. The arc current  $I_a = 100$  A.

*Variant of the anode without insert.* When the coils of the system are in accordant connection, the magnetic field throughout the length of the plasma guiding duct is mainly parallel to the walls of the latter. These are the most favorable conditions for plasma transport. The losses in this case are minimal, since the plasma resistance across the field is high and drifting of particles onto the wall is difficult. But, on the other side, in consequence of the same cause, the ignition and burning of the arc in this case is difficult too. Therefore, to ensure the reliable ignition and stable burning of the arc one have to weaken the magnetic field in the anode in front of the working surface of the cathode (Fig.4a), so that stable burning conditions were complied with the requirements given in [25]. This local weakening (“notch”) of the magnetic field takes place in the case of switching off the coils  $L_1$ . However, in our case it was insufficient. We have to connect this coil in opposite direction to the rest coils of the system, controlling the current value in it, trying to get an acceptable discharge stability. In the system under consideration it was achieved at a current  $I_1$  in the coil not less than 0.3 A. For this case the plot of the ion current  $I_i$  at the anode exit as a function of  $I_1$  is shown in Fig.5 (curve 1). A significant increase of the ion current is promoted by the opposite connection of a stabilizing coil  $L_{st}$  at raised values of  $I_1$  (more than 1A) (curve 2) when the coil  $L_4$  was connected as a correcting coil (correction by the scheme B) instead of the diagonal coil (D) (curve 3).

*The anode with the insert.* In this case for the stable ignition and burning of the arc it was not need to create the magnetic field “notch” near the cathode. Stability of the plasma generator operation was provided for any magnetic field distribution in the anode, since in the given construction the lines of the discharge current along the field lines always are short-circuited with the insert being an anode element. Therefore, it has been thought reasonable for all the coils be in accordant connection. When the “notch” is, practically, absent (Fig.4b), there is no plasma reflection from its “right bank”, and the optimum conditions for plasma loss minimization are realized on the lateral walls of the anode. The measurement results confirmed these suppositions: the ion current at the plasma generator exit was 6.5 - 8.0 A. The lateral coil  $L_4$  was used as a correcting coil (the correction scheme B).

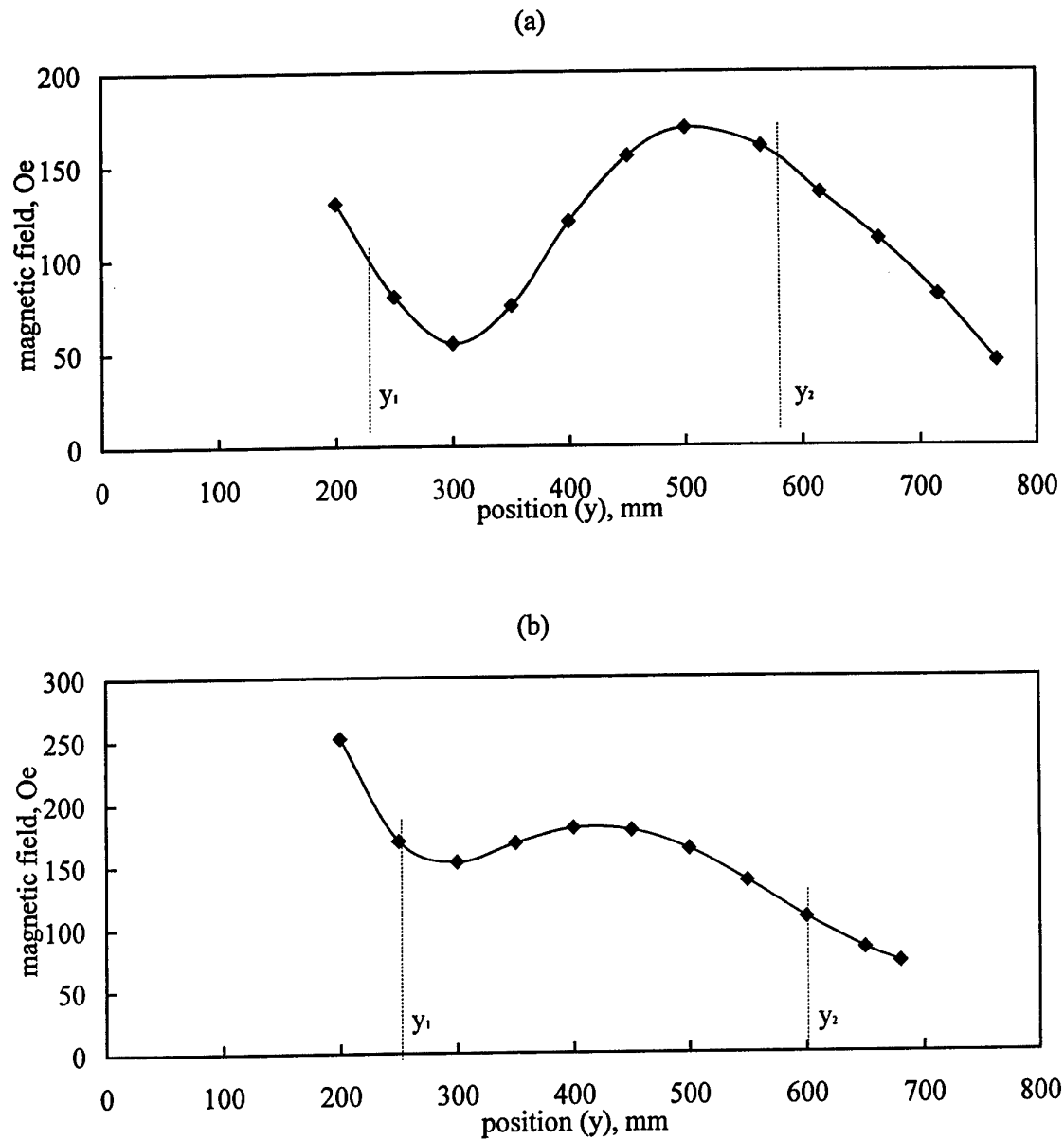


Fig. 4. Magnetic field lateral component as a function of the coordinate  $y$  along the anode axis.

(a) – without anode insert;

(b) – with anode insert.

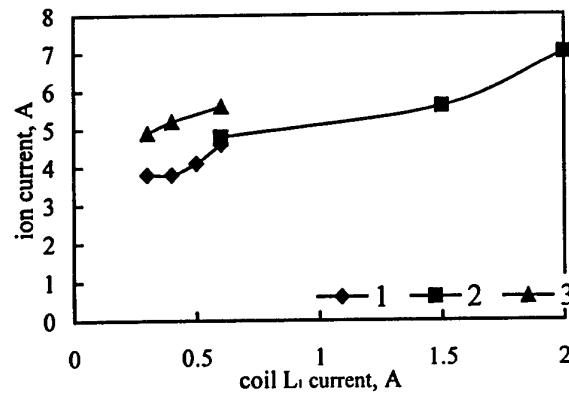


Fig. 5. Ion current at the anode exit as a function of coil  $L_1$  current.

The coil  $L_1$  is switched on oppositely to all other coils (1,3); coils  $L_{st}$  and  $L_1$  are switched on oppositely to all other coils (2). Correction of the magnetic field is realized using the schemes "D" (1,2) and "B" (3).



### 3.2. Filter transmission

#### 3.2.1. Anode without insert

The magnetic field in the plasma guiding channel of the filter is created as a result of superposition of fields from the input solenoid (focusing coils  $L_1$ ,  $L_2$  and  $L_3$ ), output solenoid (the coils  $L_7$ ,  $L_8$ ,  $L_9$ ), as well as from the correcting coils: lateral coil  $L_4$  (B), figured coils  $L_5$ ,  $L_6$  ( $F_1$ ,  $F_2$ ) and diagonal coil D (is not shown in fig.1; see Fig.2). All the coils, excluding  $L_6$ , were connected in such a manner that their fields were directed towards one and the same direction (from up to down, and from left to right). The coil  $L_6$  was either in accordant connection or in direction opposed to all other coils. The ion current onto the disk-collector and the quadrate-collector was measured when switching on the correcting coils by the schemes presented in Fig.2, by the scheme with the lateral coil B (not shown in the Fig.2) and by other combinations.

The measurements were conducted at the arc current  $I_a = 100$  A. Titanium was used as a cathode material.

Scheme “D”. Correction of the magnetic field by this scheme is a well known engineering solution [26]. Measurements of  $I_i$  with the field correction by this scheme give the control data with which the results obtained for other schemes were compared. The results of measurements for the scheme “D” are given in Fig. 6a.

The maximum current at the input into the curvilinear part of the plasma duct (up to 7A) was attained by connecting the stabilizing coil  $L_{st}$  in series opposition. The ion current at the filter exit in this case reached 1.4 A. So, the system efficiency coefficient was  $\eta = I_{iex}/I_a \approx 1.4\%$ , and the transmission coefficient was  $\theta = I_{iex}/I_{ient} \approx 20\%$ .

Using the accordant connection of  $L_{st}$  the corresponding coefficients were increased:  $\eta \approx 2.2\%$ ,  $\theta \approx 43\%$ .

Scheme “ $F_1$ - $F_2$ ”. The idea of the scheme was to create a zone with a minimum magnetic field in the region of plasma flux bending (at the joint of the input and output parts of the plasma duct). Such zone arises when coils  $F_5$  and  $F_6$  are switched on oppositely to each other. The annular slit in the magnetic field of this coils throughout the perimeter, excluding the sector on the side of the output plasma guide, is a “magnetic mirror”. On the side of the exit the slit is widened and gives a possibility for the plasma to enter freely into the output part of the filter. However, in connection with that the strength of the magnetic field in the slit from the coils  $L_5$  and  $L_6$  was found insufficiently strong, the effect expected was not attained. The ion current at the exit of such a system was rather low (Fig.7). The measurements with the use of a rotatable probe (Fig.3) showed, that the major part of the plasma flux passes through the plasma guide bend over the upper half of the plasma duct cross-section (Fig.8).

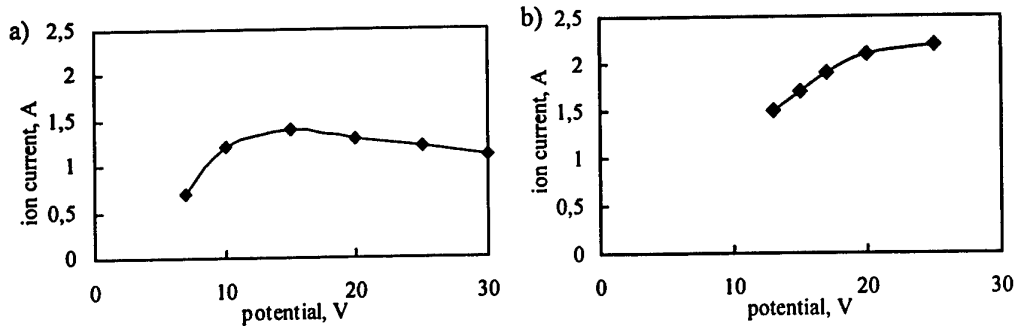


Fig. 6. Ion current of the filter exit as a function of the plasma duct potential.

(a) – the coils  $L_{st}$  and  $L_1$  are switched on oppositely to all other coils; (b) – the coil  $L$  is switched on oppositely to other coils.

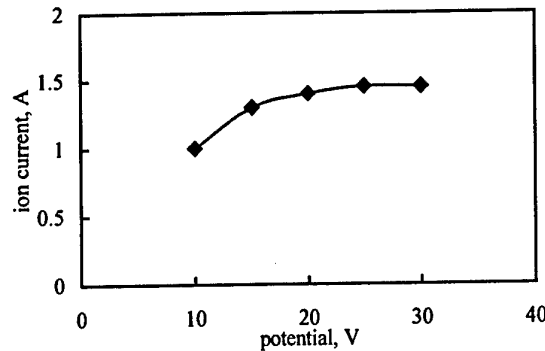


Fig. 7. Ion current at the filter exit as a function of plasma duct potential.

For correction scheme "F<sub>1</sub>-F<sub>2</sub>".

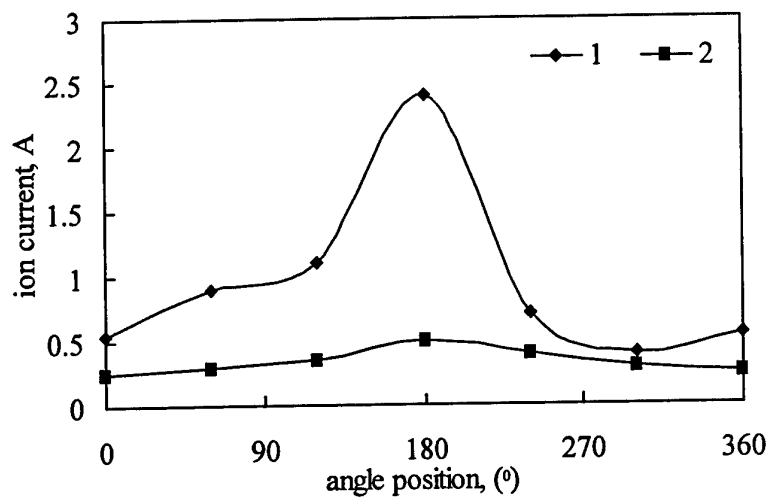


Fig. 8. Ion current of the turning probe as a function of the angle coordinate  $\alpha$ .

(1) – the output plasma duct aperture in height is overlapped by the probe completely;

(2) – the lower half of the output plasma duct is overlapped by the probe.

Scheme “D+F<sub>1</sub>-F<sub>2</sub>”. The results of measurements are given in Fig.9. Similarly to the above case,  $I_{\text{ex}}$  is not high. Its increase up to  $1.8 \div 2.6$  A at a plasma guide potential of  $14 \div 40$  V, respectively, was observed only during the short-time raise of the field strength up to 200 Oe (to avoid overheating of the coils).

The data obtained show that the idea to use a sharp-angled geometry of magnetic fields for plasma turn requires checking with the use of stronger fields.

Scheme “B + F<sub>1</sub> - F<sub>2</sub>”. The measurement demonstrated that, this variant is the more effective scheme of correction compared to all other tested variants (Fig.10). In experiments using this scheme we observed also a pronounced effect of the working cathode edge position relatively to the magnetic field “notch” (curves 1 and 2 in Fig.10, taken for the cathodes of a different length). The system coefficient  $\eta$  and the transmission coefficient  $\theta$  were 2.9% and 55%, respectively.

### 3.2.2. Anode with the insert

A grid from tungsten rods (see section 2) was used as an insert. The cathode material was titanium. The ion current was measured with the help of the disk-collector in the region of the coil L<sub>9</sub>. The results of measurement are presented in Fig.11.

Scheme “B+F<sub>1</sub>-F<sub>2</sub>”:  $\eta = (4,5 \dots 5,1)\%$ ;  $\theta = (61 \dots 70)\%$ .

Scheme “B+F<sub>1</sub>+F<sub>2</sub>”:  $\eta = (4,8\%)$ ;  $\theta = (65\%)$ .

Scheme “B + F<sub>1</sub> - F<sub>2</sub>”:  $\eta = (2,7 \dots 3,3)\%$ ;  $\theta = (3,8 \dots 4,5)\%$ .

### 3.2.3. Influence of current in correcting coils on the ion passage through the filter

Measurement of the ion current at the output of the curvilinear part of the plasma duct was performed using the disk-collector in the plane of the coil L<sub>9</sub>. The cathode material was titanium. The arc current was 100 A. The results of measurements are presented in Figs. 12-14.

The measurements were made for the correction schemes “D + B” at angles of diagonal coil inclination 45° and 15° (relatively to the horizontal plane), as well as for the scheme “B + F<sub>1</sub> - F<sub>2</sub>”.

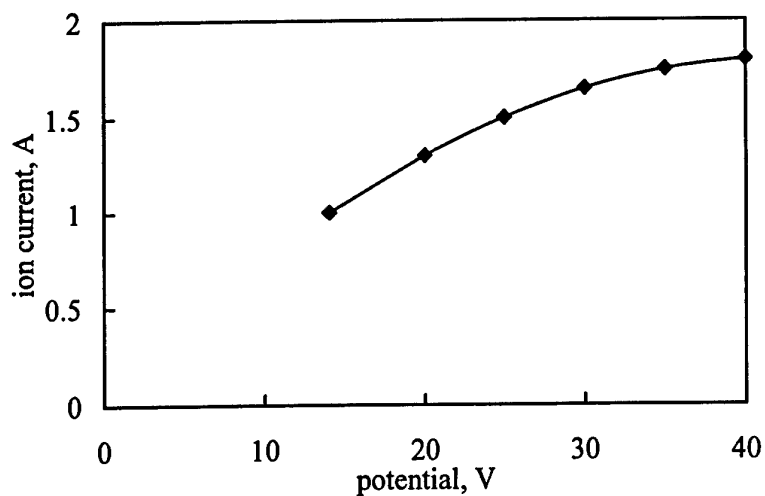


Fig. 9. Ion current at the filter exit as a function of plasma duct potential. For correction scheme "D+F<sub>1</sub>-F<sub>2</sub>".

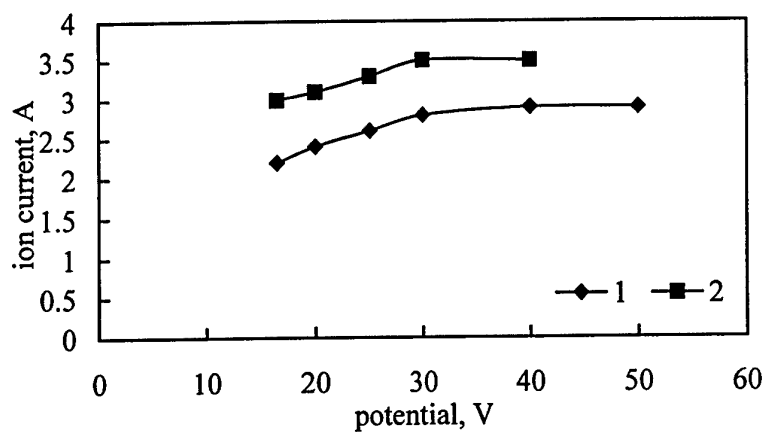


Fig. 10. Ion current of the disc-collector as a function of plasma duct potential. For correction scheme "B+F<sub>1</sub>+F<sub>2</sub>".

- (1) – for the cathode after 6 hours operation;
- (2) – for the new cathode.

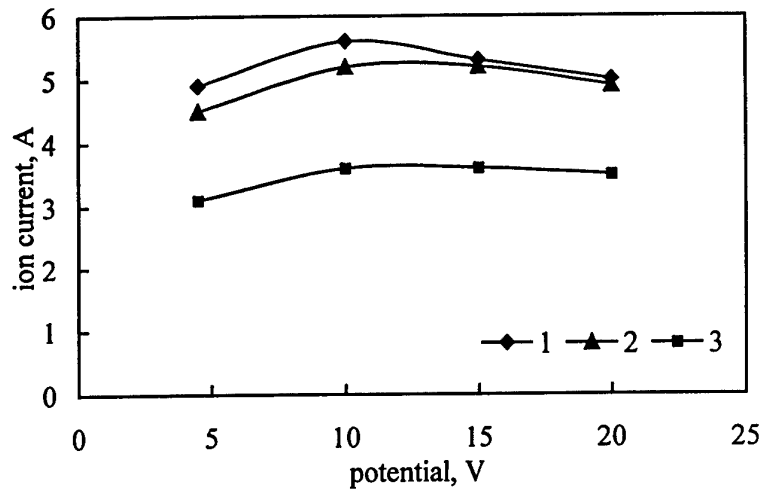


Fig. 11. Ion current of the disc-collector as a function of plasma duct potential. Anode with insert. For correction schemes "B+F<sub>1</sub>-F<sub>2</sub>" (1), "B+F<sub>1</sub>+F<sub>2</sub>" (2), "F<sub>1</sub>-F<sub>2</sub>" (3).

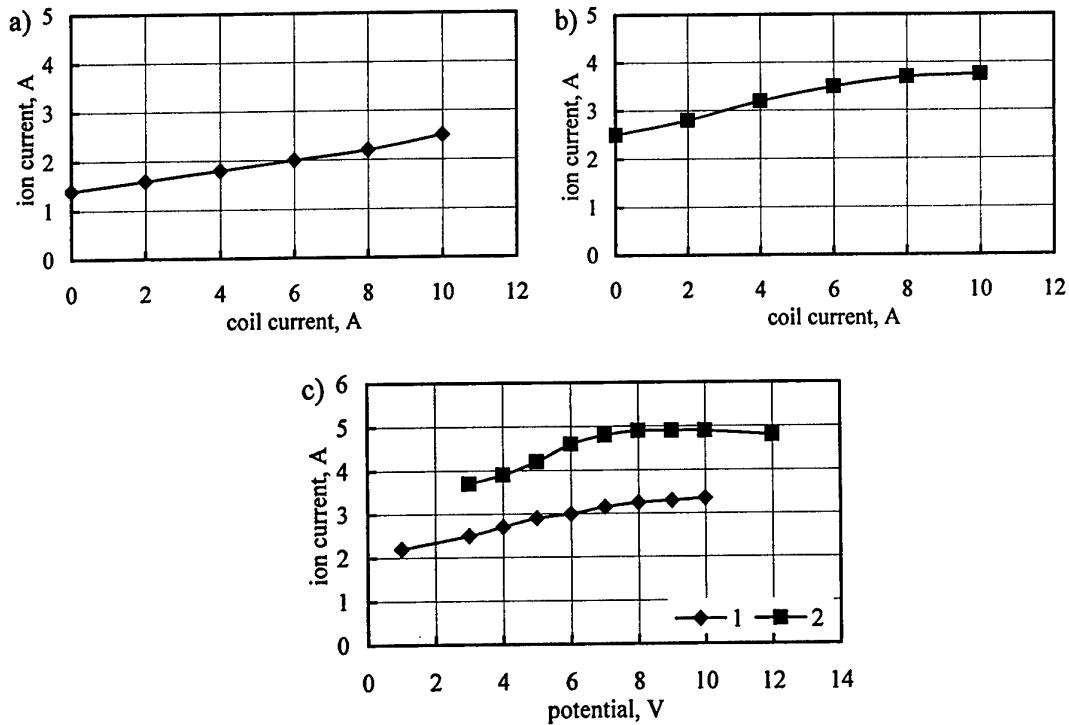


Fig. 12. Ion current of the disc-collector as a function of currents in the diagonal (a) and lateral (b) coils and plasma duct potential (c). Diagonal coil slope angle is 45°.

(a) – lateral coil current  $I_B$  is 0;

(b) – diagonal coil current  $I_D$  is 10 A;

(c) –  $I_D = 10$  A,  $I_B = 0$  (1);  $I_D = I_B = 10$  A (2).

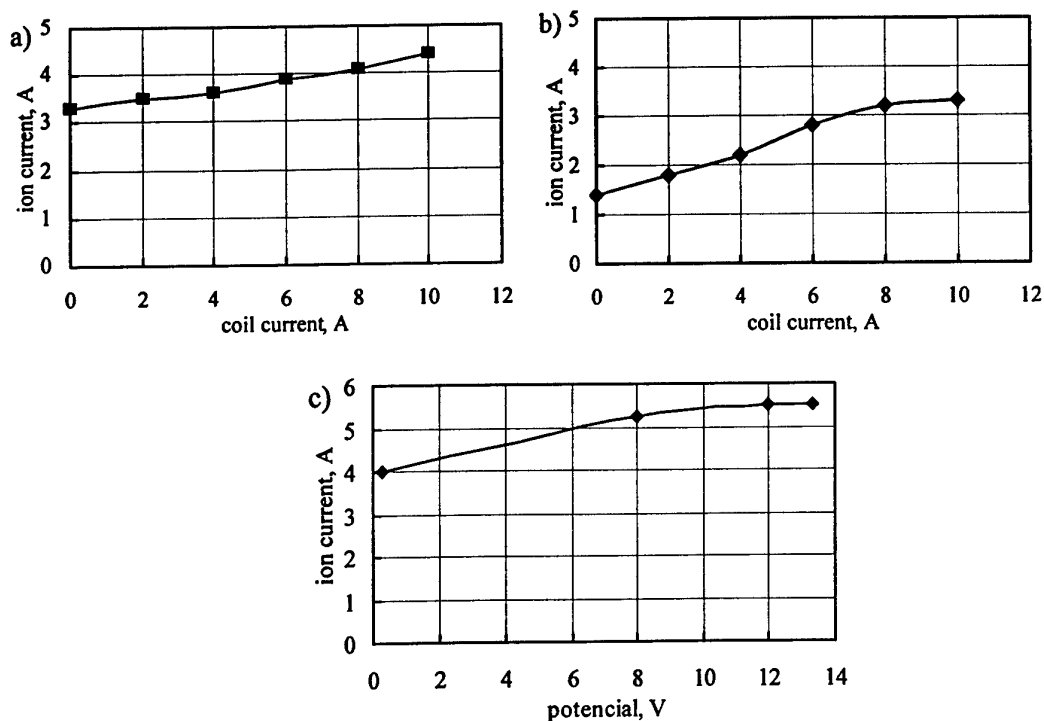


Fig. 13. Ion current of the disc-collector as a function of current in diagonal (a) and lateral (b) coils and plasma duct potential (c). Angle of diagonal coil slope is  $15^\circ$ .

$I_B = 10$  A (a);  $I_D = 0$  (b);  $I_B = 10$  A,  $I_D = 8$  A (c).

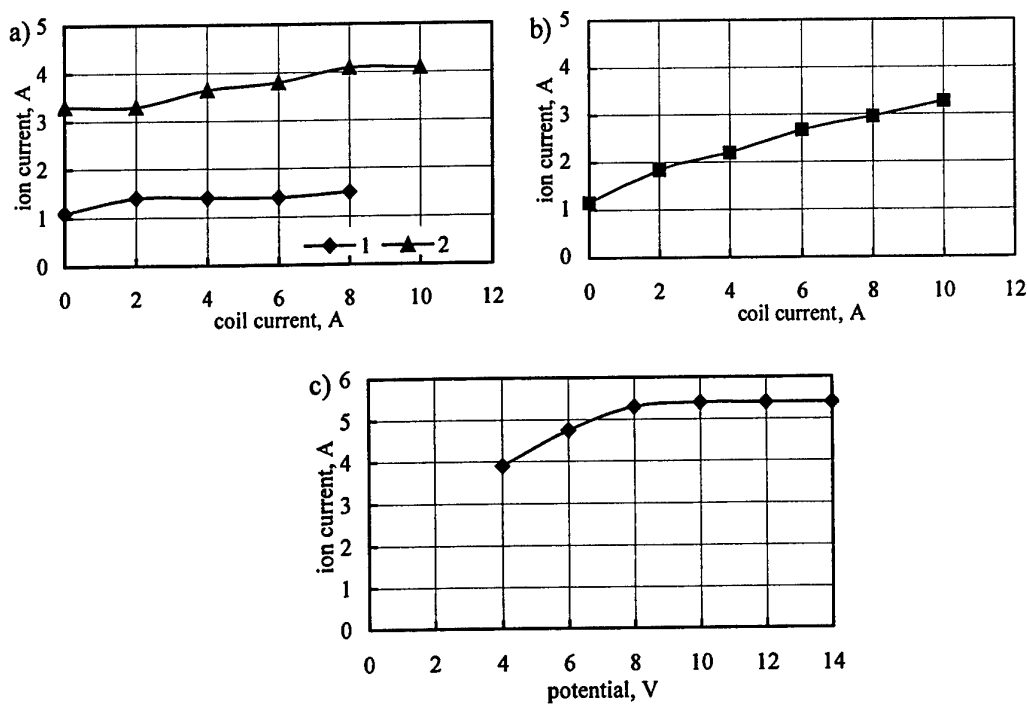


Fig. 14. Ion current of the disc-collector as a function of current in figured (a) and lateral (b) coils and plasma duct potential (c).

(a)  $I_B = 0$  (1),  $I_B = 10$  A (2); (b)  $I_F = 0$ ; (c)  $I_B = I_F = 10$  A.

#### 4. EXPERIMENTS WITH THE GRAPHITE CATHODE

As it was noted earlier in Ref. [22] in the case of the vacuum arc with a graphite cathode there are difficulties in ignition and stable burning of the discharge at pressures  $p \leq 10^{-5}$  Torr. Stability of the discharge increases significantly when one uses the anode insert and argon feed into the system to the pressure  $p = (7\div 9) \cdot 10^{-5}$  Torr. We have tested the anode inserts of two types. In the first variant it was a graphite plate-disk of 6 mm in thick, 170 mm in diameter, with 19 orifices of 22 mm in diameter uniformly distributed in the central part of the disc. The tests have shown that after 20 min discharge burning at an arc current of 110÷130 A in the bridges between the orifices the through cracks there were arised and, as a result, the discharge become instable, and we had to stop the experiment.

In the second variant the anode insert was made as a set of plates from the carbon cloth impregnated with pyrocarbon (see section 2). To remove thermal stresses the plates were fixed on the copper compensators being the current leads. This construction demonstrated a good mechanical strength.

The ion current at the filter exit was measured using the circuit of the correcting magnetic field " $B + F_1 - F_2$ " at  $I_a = 110$  A. The collector was disposed in the plane of the output coil  $L_9$ . When the plasma guide potential was changing from +8 V to + 20 V the ion current onto the collector was 2.2÷4 A. To ensure the stable burning of the discharge the system was filled with argon up to a pressure of  $(5\div 6) \cdot 10^{-5}$  Torr.

The ion density profile in the flow was determined by the rate of depositing the carbon coating at the filter exit. For measurements we used a polished plate from the stainless steel of 235x235x1 mm<sup>3</sup> as a collector . The collector was placed in the chamber at distances 45, 120 and 180 mm from the filter exit. The current onto the collector was 1.5÷2 A. The exposition time was 90 s, 150 s, and 90 s, respectively. The results of measuring the deposition rate distribution along the horizontal X and vertical Y axes are given in Figs. 15, 16 and 17. Maximum values of the deposition rate were 18, 13 and 8.5  $\mu\text{m/h}$  at distances 45, 120 and 180 mm from the filter exit, respectively. The nonuniformity of the coating thickness does not exceeds 4% on the diameter of 5÷6 cm. The deposition rate is maximum in the region having the shape close to the circle. The center of this region is displaced upwards (along Y) by 0.5 and 3 cm for the collector location from the filter exit 120 and 180 mm, respectively. Displacement along the axis X with distance changing occurs nonmonotonously (-4 cm; -0.5cm; -5 cm). Probably, it is caused by the low velocity and nonregular motion of the cathode spot in the process of film deposition.

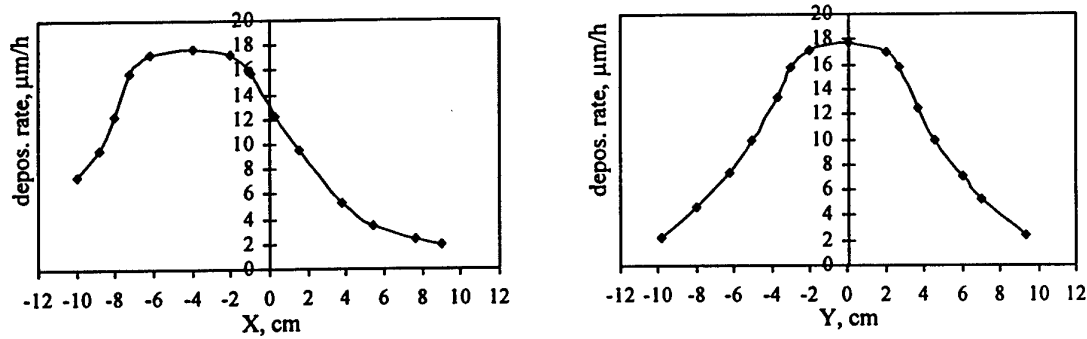


Fig. 15. Distribution of DLC deposition rate along the horizontal (x) and vertical (y) axis. A substrate is located at the distance from the filter exit end of 4,5 cm. Y axes overpasses horizontal axes (x) at  $x = -4$  cm.

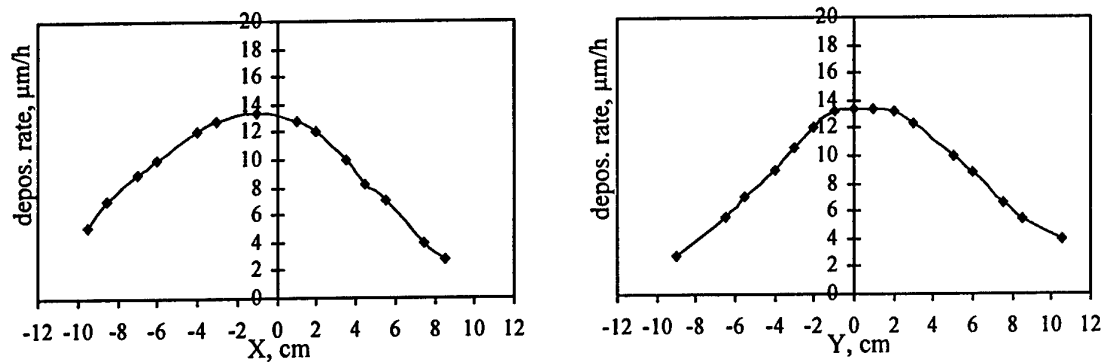


Fig. 16. Distribution of DLC deposition rate along the horizontal (x) and vertical (y) axis. A substrate is located at the distance from the filter exit end of 12 cm. Y axes overpasses horizontal axes (x) at  $x = -0,5$  cm.

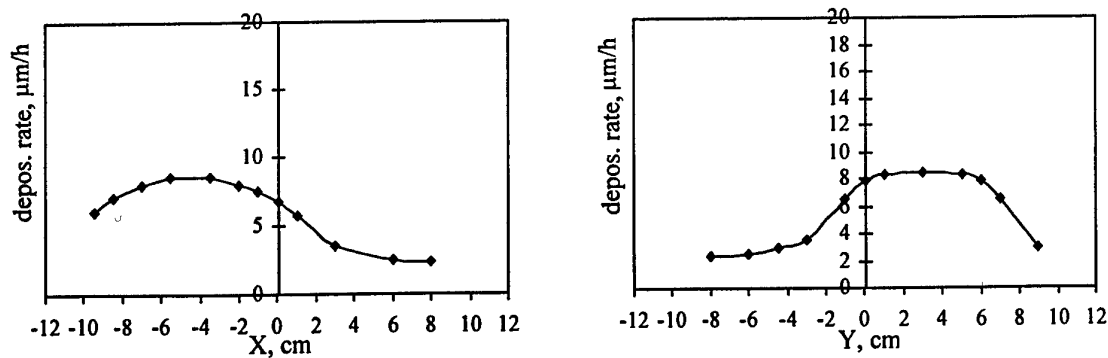


Fig. 17. Distribution of DLC deposition rate along the horizontal (x) and vertical (y) axis. A substrate is located at the distance from the filter exit end of 18 cm. Y axes overpasses horizontal axes (x) at  $x = -5$  cm.



## 5. COMPARATIVE STUDY OF DLC AND CN<sub>x</sub> COATINGS PRODUCED BY PULSED LASER AND FILTERED VACUUM ARC TECHNIQUES

Unhydrogenated amorphous diamond-like carbon (DLC) and fullerene-like carbon nitride (CN<sub>x</sub>, N/C ratio ~0.2) are rival surface protection coatings with exceptional mechanical and tribological properties. A comparative study was performed on their benefits for sliding wear protection. The coatings were grown in the same deposition system, using similar substrates, surface preparation procedures, adhesive interlayer (Ti-Ti<sub>1-y</sub>C<sub>y</sub>,  $y$  graded from 0 to 1), and top layer thickness. They were produced using pulsed laser deposition (PLD) and filtered cathodic arc deposition (FAD). Samples of laser-DLC, laser-CN<sub>x</sub>, arc-DLC, and arc-CN<sub>x</sub> coatings were prepared. Comparisons of coating chemistry, structure, hardness, elastic modulus, internal stress, coefficient of friction (c.o.f.) against steel and SiC balls, wear rate, and wear mechanism in humid and dry environments were performed. The comparison indicates that PLD and FAD provide very similar coating chemistry, structure and properties. Independent of the growth technique. DLC coatings had hardnesses within 52-57 GPa and elastic moduli within 490-560 GPa. The CN<sub>x</sub> coatings offered a reasonably high hardness of 28-30 GPa, while their elastic modulus was as low as 160 GPa. There was a clear difference in tribological behavior of DLC and CN<sub>x</sub>, which depends on the environment humidity. In humid air, DLC coatings had a c.o.f. of 0.1, a very low wear rate, and formed a graphitic transfer film in friction contact. In the same tests, CN<sub>x</sub> coatings had a c.o.f. of 0.3-0.4, a higher than DLC wear rate, and did not form a transfer film. In this environment, wear tracks on CN<sub>x</sub> coatings were polished by abrasion wear. In dry nitrogen, DLC coatings had a c.o.f. of about 0.15 and a higher wear rate, while CN<sub>x</sub> coatings had a c.o.f. 0.03-0.04 and a lower wear rate with formation of a graphitic-like transfer film. The observed difference in mechanical response and tribological performance can be used to optimize selection between DLC and CN<sub>x</sub>, depending on application requirements.

## 6. CONCLUSIONS

As it follows from the data of the present report, and the annual report [22], the main results of the works under the project P-069 consist in the following.

- 1) It is shown experimentally, that the magnification of linear dimensions of the plasma duct aperture of the magnetic filter (up to 200...300 mm) and lowering of the relation of its radius of curvature to radius of its aperture promotes considerable increase of throughput capacity of the system. The basic losses of plasma at its passage through the filter pertain to the curvilinear part of the plasma duct. Here losses are stipulated by a diffusion of plasma crosswise of transporting magnetic field, and also by shifts of the plasma stream to plasma duct walls owing to gradient and centrifugal drifts. It is found out, that the drift losses can be appreciably reduced by correction of a magnetic field in the curvilinear part of the plasma guiding channel. The level of losses, irrespective of the mechanism of their origin, is reduced, and the ion current on the exit of the filter increases with rise of positive bias potential of the duct. The shift of a main body of the plasma stream in the plane of symmetry of the duct occurs in a streamline of a leading lines of a magnetic field, which intercross working surface of the cathode. In explored system these magnetic lines and, hence, the guided plasma stream shifted to centre of the duct curvature, while the theories of other researchers order shift of the stream in an opposite direction.
- 2) The obtained results of experimental examinations and calculation are utilised for development of the concept of the new high-performance system of a filtrated plasma flow forming. The system, designed and manufactured according to this concept, contains the vacuum-arc generator of plasma with magnetic stabilization of a cathode spot motion zone and with magnetic focusing of a plasma stream generated by this spot. The tubular anode with focussing coils executes also functions of an input arm of the knee-shaped filter with a bend of a plasma stream on  $90^\circ$ . The plasma duct comprises a trap of macroparticles having form of cylindrical container, which walls are the continuation of walls of the input plasma duct, so the filter as a whole acquires the T-shaped design. The filter is equipped with a transition section for linking with the chamber of laboratory installation in AFRL. To insure of reliable ignition and stable burning of an arc the grid-like insert is disposed inside the anode.
- 3) Some variants of arrangement and feed circuits of coil for correction of a magnetic field distribution in the plasma guiding duct bending are explored. In optimum variant the ion current of up to 5,6 A at the exit of a curvilinear part of the plasma duct is obtained. The system effectiveness ratio was 5,1% (Ti cathode), that is much higher, than for other known sources with the filter of a similar type.

- 4) In experiments with the graphite cathode DLC film deposition rates of 18, 13 and 8,5  $\mu\text{m/h}$  on distances 45, 120 and 180 mm from exit of the filter are obtained. On a site of a substrate of 60 mm in diameter the inhomogeneity of film thickness distribution did not exceed 4%.
- 5) Hydrogen-free amorphous diamond-like carbon and fullerene-like carbon nitride were produced in AFRL using pulsed laser deposition and filtered cathodic arc deposition based on the new filtering system in question. The films obtained by these two methods are similar to each another.
- 6) The results of R&D under the project P-069 are described in the Refs. [27...30] and are reported at international conferences [31,32].

## REFERENCES:

1. P.J.Wilbur, Dual-use application of ion-thruster technology, in: *Space Technology and Applications International Forum (STAIF-96); Proceedings of the 1<sup>st</sup> Conference on Commercial Development of Space*, Albuquerque, NM, Jan/ 7-11, 1996. Pt. 1 (A96-19865 04-83), Woodbury, NY, American Institute of Physics, *AIP Conference Proceedings*, 361 (1996) 155-161.
2. S.Aisenberg and R.Chabot, Ion-beam Deposition of Thin Films of Diamondlike Carbon, *J. Appl. Phys.*, 42 (1971) p. 2953; S.Aisenberg and R.Chabot, *NASA Electronics Research Center Report* (Contract NAS 12-541), 1969.
3. A.A.Voevodin, M.S.Donley and J.S.Zabinski, Pulsed Laser Deposition of Diamond-Like Carbon Wear Protective Coatings: A Review, *Surface and Coating Technology*, 92 (1997) 42-49.
4. I.I.Aksenov, V.A.Belous, S.I.Vakula, V.G.Padalka, V.E.Strel'nitskij, V.M.Khoroshikh, High-efficiency source of pure carbon plasma, *Sov. Phys. Tech. Phys.*, 50 (9) (1980), 2000-2004.
5. A.S.Bakaj, V.E.Strel'nitskij. Structural and physical properties of carbon films prepared by deposition of fast particle flows. – Review. – M.: TsNIIatominform, 1984, 87 p.
6. A.S.Bakaj, A.V.Baranov, V.E.Strel'nitskij, Investigation of carbon films structure by Raman spectroscopy, *Sov. Surface*, 3 (1990) 92-98.
7. I.I.Aksenov, V.E.Strel'nitskij. Wear resistance of diamond-like carbon coatings, *Surface and Coating Technology*, 47 (1991) 252-256.
8. I.I.Aksenov, V.E.Strel'nitskij, Properties of diamond-like coating prepared by vacuum-arc depositions, *Surface and Coating Technology*, 47 (1991) 98-105.
9. I.I.Aksenov, M.G.Maksimov, Yu.Ya.Palij, V.E.Strel'nitskij, Tribological behavior of diamond-like coatings at high rates of sliding, *Diamond and Related Materials*, 2 (1993) 866-868.
10. I.I.Aksenov, V.A.Belous, V.E.Strel'nitskij, N.V.Samokhvalov, O.A.Opalev, Some results obtained by the Kharkov research group in the area of development highly efficient methods of diamond coating deposition, "Eurodiamond '96", *Conference Proceedings*, vol. 52, C.Manfredotti and E.Vittone (Eds), SIF, Bologna (1996) 17-26.
11. I.I.Aksenov, V.A.Belous, V.G.Padalka, V.M.Khoroshikh, Plasma filtering device, , *Instrum. Exper. Tech.*, 5 (1978) 236-237.
12. I.I.Aksenov, V.G.Padalka, V.M.Khoroshikh, Investigation of plasma flux generated by a steady-state erosion arc accelerator with magnetic cathode spot confinement, *Sov. J. Plasma Phys.*, 5 (3) (1979) 607-612.
13. I.I.Aksenov, N.S.Repalov, V.G.Padalka, V.M.Khoroshikh, Investigation of plasma flux motion in a toroidal plasma guide, *Sov. J. Plasma Phys.*, 6 (2) (1980) 312-318.
14. I.I.Aksenov, V.G.Padalka, V.T.Tolok, V.M.Khoroshikh, Investigation of vacuum arc plasma flux motion in a rectilinear plasma guide, *Sov. J. Plasma Phys.*, 6 (4) (1980) 918-924.
15. I.I.Aksenov, V.G.Bren, V.G.Padalka, V.M.Khoroshikh, Mechanism of shaping the ion energy distribution in the plasma of vacuum arc, *Sov. Tech. Phys. Lett.*, 7 (19) (1981) 1164-1167.
16. I.I.Aksenov, A.N.Belokhvostikov *et.al*, Plasma flux motion in a toroidal plasma guide, *Plasma Physics and Controlled Fusion*, 28 (5) (1980) 761-770.
17. I.I.Aksenov *et.al*, Arc plasma generator and a plasma arc apparatus for treating the surfaces of work-pieces incorporating the same arc plasma generator, *Canadian Patent №1176599*, Oct. 1984.
18. I.I.Aksenov *et.al*, Vacuum-arc plasma apparatus. – *US Patent №4551221*, Nov. 1985.
19. B.F.Coll, *Proc. Of the 4<sup>th</sup> Int. Symp. On Trends and New Appl. In Thin Films – TATF'94*, Dresden, (1994) 3-17.
20. R.L.Boxman *et.al*, Recent progress in filtered vacuum arc deposition. *Surf. Coat. Technol.*, 86/87 (1996) 243-253.
21. A.Anders, Approaches to rid cathodic arc plasmas of macro- and nanoparticles: a review. *Surf. Coat. Technol.*, 120/121 (1999) 319-330.

22. Magneto-Optically and Fully Ionised Metal/Carbon Arc Plasma Source for Thin Film Coating to Control, Wear and Fretting. Science and Technology Center in Ukraine, *Project P-069, Annual report*, NSC KIPT, Kharkiv, 2001.
23. I.I.Aksenov, V.A.Belous, Vacuum arc excitation in metal plasma sources. *Teplofizika vysokikh temperatur*. V.17, №1, 1979, p.p. 1-4 (Rus.).
24. I.I.Aksenov, V.A.Belous, Vacuum arc ignition in d.c. sources of metal plasmas using an autonomous plasma injector. *Priboryi tehnika experimenta*, №3, 1979, p.p. 160-162 (Rus.).
25. I.I. Aksenov (Aksenov), V.G. Bren, V.G. Padalka, V.M. Khoroshikh, Vacuum-Arc Plasma Apparatus. US Patent №4551221, Nov. 5, 1985; Filled Mar. 2, 1981.
26. I.I.Aksenov, D.Yu. Zaleskij, and V.E. Strel'nitskij. On Efficiency of Systems for Filtered Cathodic Arc Deposition, 1<sup>st</sup> International Congress on Radiation Physics, High Current Electronics, and Modification of Materials, Tomsk, Russia, 24-29 September 2000. Proc. Vol. 3, p.p. 133-138.
27. I.I. Aksenov, V.E. Strel'nitskij, Synthesis of hydrogen free DLC films: review // Proceedings of 12<sup>th</sup> International Symposium "Thin film in electronics", Kharkov, Ukraine, April 24...27, 2001, p.p. 96-105.
28. I.I. Aksenov, D.Yu. Zaleskij and V.E. Strel'nitskij, On the efficiency of plasma filters for the cathodic arc // Proceedings of the Sixth Applied Diamond Conference/Second Frontier Carbon Technology Joint Conference (ADC/FCT 2001), Auburn, Alabama, USA. 2001, p.p. 509-515.
29. I.I. Aksenov, V.E. Strel'nitskij, Vacuum-arc discharge as an instrument for PVD process of DLC films deposition // Proceedings of the 5<sup>th</sup> International Symposium "Diamond films and films of diamond-related materials", 22-27 April, Kharkov, Ukraine, 2002, p.p. 39-64.
30. I.I. Aksenov, V.V. Vasilyev, D.Yu. Zaleskij, Efficiency of magnetic plasma filters // Surface and Coatings Technology. Vol.163-164, 2003, p.p. 118-127.
31. I.I. Aksenov, V.E. Strel'nitskij, High quality diamond-like carbon films synthesized by the vacuum-arc method. Int. Conf. "Science and Technology of Carbon and Carbide Materials - 2002", February 11-13, 2002, La Jolla, Ca., USA.
32. I.I. Aksenov, V.E. Strel'nitskij, V.V. Vasilyev, D. Yu. Zaleskij. Efficiency of magnetic plasma filters, Int. Conf. on the Metallurgical Coatings and Thin Films, ICMCTF 2002, April 20-27, 2002, San Diego, Ca., USA.